TECHNICAL REPORT 3287

EVALUATION OF INORGANIC NITRATES AS HEAT TEST STANDARDS

ICRPG WORKING GROUP ON ANALYTICAL CHEMISTRY

ROUND ROBIN NO. 24

MILTON ROTH

COPY 67 OF 72

DECEMBER 1955

PICATINNY ARSENAL
DOVER, NEW JERSEY

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BY

MILTON ROTH

DECEMBER 1965

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ACKNOWLEDGMENT

The author is grateful to Vincent K. Canfield and Robert E. Young of Picatinny Arsenal's Feltman Research Laboratories' Analytical Chemistry Branch for their assistance in testing the large number of inorganic nitrates that provided the basic data needed to initiate this round robin.

SUMMARY

The data from this round robin indicates that aluminum nitrate, $Al(NO_3)_3 . 9H_2O$, and copper nitrate, $Cu(NO_3)_2 . 3H_2O$, are suitable for use as heat test standards. These compounds are preferable to the organic nitrates currently being used because of greater stability, safety, purity and availability. These findings were reported at the 22nd meeting of the Interagency Chemical Rocket Propulsion Group (ICRPG) on Analytical Chemistry at the NASA-Lewis Research Center in Cleveland on 3-5 November 1965.

CONCLUSIONS

$Al(NO_3)_3 . 9H_2O$ and $Cu(NO_3)_2 . 3H_2O$ are suitable for use as standards in the potassium iodide (KI) and methyl violet (MV) heat tests.

In addition to being more stable than the organic nitrates currently used as standards, these inorganic nitrates are safer to store and easier to obtain at a prescribed level of purity.

Standardization of the heat test can be improved if inorganic nitrates are distributed with the test papers along with a statement of the test time at a given temperature.

RECOMMENDATIONS

The method of standardizing KI-starch and MV heat test papers should be based on inorganic nitrates (such as Cu or Al) rather than nitrocellulose or nitrocellulose base propellants.

The inorganic nitrate standard should be distributed along with the test papers. The time required for the papers to respond at a given temperature should be stated. This combination will provide a reference that will enable all laboratories to adjust their equipment so that better agreement is attained.

The inorganic nitrate standard should be used by the laboratories at periodic intervals to assure that no change occurred in their testing equipment or procedure.

The inorganic nitrate standard should be used to train new operators.

Action Taken

The results of this round robin were reported at the 22nd meeting of ICRPG Working Group on Analytical Chemistry at the NASA-Lewis Research Center in Cleveland on 3-5 November 1965.

The U.S. Naval Propellant Plant was requested by means of this report to use the inorganic nitrates to standardize the test papers which are distributed for use in this test.

BACKGROUND

During the course of work on a project designed to improve the objectivity of the MV and KI heat tests, it was found that these tests also were applicable to large number of inorganic nitrates (Reference 1). Since inorganic nitrates at high levels of purity are commercially available and are much more stable than the organic nitrates which are now used as heat test standards, it appeared desirable to investigate the possibility of replacing the current standards with the inorganic analogs in order to minimize one of the most serious shortcomings of these tests.

The heat tests are dependent on the assumptions that neither the organic nitrate standard (NC or related propellant) nor the test papers change with time. It is particularly important for the validity of these tests to have a stable standard but organic nitrates are known to be deficient in this property. By substituting inorganic nitrates the reliability of the heat tests should be greatly improved.

For this reason, a round robin was organized with the cooperation of the following laboratory members of the ICRPG Working Group on Analytical Chemistry:

E.I. du Pont -- Carney's Point, New Jersey

Hercules Powder Co. --

Alleghany Ballistics Laboratory -- Cumberland, Maryland

Kenvil Plant -- Kenvil, New Jersey

Olin Mathieson Chemical Co. -- East Alton, Illinois
Marion, Illinois

Frankford Arsenal -- Philadelphia, Pennsylvania

U.S. Naval Propellant Plant -- Indian Head, Maryland

U.S. Naval Weapons Station -- Concord, California

Picatinny Arsenal -- Dover, New Jersey

Statistical analysis of the data by the usual analysis-of-variance technique was inapplicable to the MV results because they are incremental rather than variable. (The results are reported to the nearest five minutes rather than to the nearest minute.) With the KI paper, statistical analysis was not applied because statistical significance is not related to practical significance. In both tests, the interpretation of the data is based on knowledge of the properties of the existing standards and the variability contributed by equipment and by test papers.

DISCUSSION OF RESULTS

The data received was classified and combined into these tables:

Table	Contents
1-4	KI Heat Test Values
5-8	MV Heat Test Values for Salmon Pink Color
9-12	MV Heat Test Values for Red Fumes
13	Summary of Heat Test Values for Al and Cu

In Tables 1-12, the replications are designated a, b and c and the average of the results is designated by $\bar{X}$. All the replications are listed as reported by the individual laboratories but the averages are rounded to the nearest minute for the KI test and to the nearest five minutes for the MV test since these are the units in which the results are customarily reported. Although four compounds were evaluated in this round robin, the data shows that only the $Al(NO_3)_3.9H_2O$ and the $Cu(NO_3)_2.3H_2O$ respond in a reproducible manner. Results for these two compounds (summarized in Table 13) show about the same reproducibility in each case. It also is evident that the materials designated as standards (compounds provided by the round robin chairman) show somewhat better reproducibility than the samples (compounds provided by cooperating laboratories).

Results compare favorably with these in ICRPG Round Robin No. 10 in which different lots of MV paper were compared (Reference 2). In Round Robin No. 10, it was found that a significant difference did exist between laboratories; subsequently a study of the equipment used by the various laboratories showed wide variations in well dimensions and rate of heat transfer (Reference 3). Since the same equipment probably is still in use and is now almost 10 years older, the reproducibility of values obtained with $Al(NO_3)_3.9H_2O$ and $Cu(NO_3)_2.3H_2O$ is indeed impressive.

For the KI test, the $Al(NO_3)_3.9H_2O$ seems slightly preferable to the copper salt at 82° while the reverse is true at 65°. For the MV test, the salts appear equally reproducible at 120° while the copper salt is slightly better at 134.5°. It is interesting to note that with the MV test, there is little difference in results or reproducibility between the so-called "standards" and "samples."

DESIGN OF ROUND ROBIN NO. 24

Round Robin No. 24 was designed so that a measure of reproducibility was obtained for the laboratories as well as the materials. Four samples were distributed by the round robin chairman and were designated as the standards for that particular salt. In addition, the laboratories were directed to test the same four nitrates or samples obtained from their stock and to designate these as samples. A comparison of the results with the material designated as "sample" with those designated as "standard" gave an estimate of the difference between materials within a laboratory. The material designated as "standard" gave an estimate of the interlaboratory reproducibility.

Detailed instructions for conducting the round robin and the data sheets that were developed are in Appendix A. The remarks submitted by the various laboratories are recorded in Appendix B.

TEST METHODS

Potassium Iodide (KI) Heat Tests (65.5° and 82.2°C)

Transfer three 2.5 gm samples to individual glass test tubes (pyrex or equivalent) that are each 5.5 inches long, 0.5 inches ID and 0.62-inch OD. Stopper each tube with a cork through which passes a glass rod equipped with a platinum holder for a strip of standard KI starch indicator paper. Attach a strip of KI paper (1-inch long, 3/8-inch wide) to the holder by means of forceps. (Do not touch the paper with your hands.) Moisten a horizontal section in the upper half of the paper with a 50% solution (v/v) of glycerin and water by drawing a line with the aid of a small diameter glass rod which has been dipped into this solution. The lower edge of the moistened line shouldbe straight and distinct across the paper. Prepare a blank tube for concurrent testing.

Adjust the temperature of the bath to within 1°C of the test temperature. Insert the test paper so that the lower edge of the moistened line is three inches above the sample and at an equal height in the blank tube. Insert all four tubes into the bath to a depth of about two inches. Note the time of insertion and the time required (to the nearest minute) for the first appearance of any discoloration at the line of demarcation between the wet and dry portion of the test paper in the sample tube which exceeds that observed in the blank tube. For optimum viewing the tubes should be placed against a white background and illuminated with bright, diffused daylight.

Methyl Violet (MV) Heat Tests (120° and 134.5°C)

Transfer three 2.5 gm samples to individual glass tubes (pyrex or equivalent) that are 290mm long, 15mm ID and 18mm OD. Into each tube place a strip of the standard MV test paper so that the lower end of the paper is 25mm from the top of the sample. Stopper each tube with corks containing holes 4mm in diameter or notches of equivalent area.

The constant temperature apparatus used to the test should be capable of maintaining the desired temperature ±0.5°C. The diameter of each thermowell in the bath should be 19 ±0.5mm and the depth should be 283.5 ±0.5mm. Insert all three tubes into the bath and note the time. When five minutes less than the expected time has elapsed (as determined by preliminary tests) quickly lift the tube half-way out of the thermowell and note whether the test paper has changed completely to salmon pink. If the change is incomplete continue the heating and recheck at five-minute intervals. Record the time required for completion of the test in each tube.

REFERENCES

1. M. Roth, Inorganic Nitrates as Standards in the Methyl Violet and Potassium Iodide Heat Tests, Picatinny Arsenal Technical Report 3179, July 1964.

2. R. H. Pierson, Cooperative Test Projects of the Joint Army-Navy-Air Force Panel on Analytical Chemistry of Solid Propellants, Part 6, U.S. Naval Ordnance Test Station Report 1937, February 1958.

3. J. L. Myers, Report on Cooperative Programs to Check Thermal Characteristics of the 134.5°C MVP Bath, Canadian Armament Research & Development Establishment Technical Letter 1064/57, Valcartier, Quebec, Canada, October 1957.

APPENDICES

APPENDIX A

INTERAGENCY CHEMICAL ROCKET PROPULSION GROUP. WORKING GROUP ON ANALYTICAL CHEMISTRY

INTERAGENCY CHEMICAL ROCKET PROPULSION GROUP WORKING GROUP ON ANALYTICAL CHEMISTRY

SUBJECT: Evaluation of Inorganic Nitrates as Heat Test Standards

TO: Participants in Round Robin No. 24

1. Inclosed herewith are the following materials for use in subject round robin:

a. Four samples of inorganic nitrates.

b. Special instructions for collecting and reporting data.

c. Data sheets (in duplicate).

d. Methods for potassium iodide (KI) and methyl violet (MV) heat tests.

2. It is requested that the completed data sheets be returned to the undersigned no later than 1 December 1964.

3. The code number for your laboratory is _____.

Very truly yours,

MILTON ROTH
Picatinny Arsenal
Process Engineering Laboratory
Building 355
Dover, New Jersey 07801

SPECIAL INSTRUCTIONS FOR DATA SHEETS

Data Sheets

The data sheets are provided in duplicate, but only one copy of the completed sheets needs to be returned to the chairman of this round robin at the following address:

Milton Roth
Picatinny Arsenal
Process Engineering Laboratory
Dover, New Jersey 07801

Completed data sheets are due by 1 December 1964. Report heat test values for each tube of each sample. In the MV test, continue heating beyond the color change until red fumes appear. If red fumes are not obtained within five hours, report 300+ minutes.

Report all values for each sample (see rejection of data).

Test Plan

In addition to testing the submitted samples, each laboratory should obtain and test samples of similar grade and granulation. This will afford measures of variability between laboratories and between samples for the same material. In all cases use the same test papers, baths and glassware as in the established methods.

On Data Sheet 4, give a complete description of the chemical and physical properties of the compounds supplied by you for this round robin. Include name of manufacturer, grade, limits of impurities and granulation. Save your samples so that, if requested, rechecks can be made.

Rejection of Data

If sample values do not agree within a few minutes, continue testing until checks are obtained. Report all values obtained.

Order of Run

The tests should be randomized in order of run. For example, each test can be assigned a number and a group of these numbers can be drawn at random, depending on the number of tests to be done on a given day.

Remarks

Space is provided on the data sheets for comments, suggestions or observations concerning the tests. If the space is insufficient, additional sheets may be added.

Samples

The following samples are inclosed:

Compound	Fisher Catalog Number	Granulation, % on			Designation
		#12	#30	#40	
$Al(NO_3)3.9H_2O$	A-586	68	225	3	Al
$Co(NO_3)_2.6H_2O$	C-378	34	65	1	Co
$Cu(NO_3)_2.3H_2O$	C-467	33	54	7	Cu
$Ni(NO_3)_2.6H_2O$	N-62	6	78	11	Ni

Sieve numbers (#) correspond to openings of 1.68-, 0.595- and 0.420mm. As indicated above, these compounds were obtained from the Fisher Scientific Company and represent the company's Certified Grade. These are designated as standard (Std) on the data sheet. Compounds furnished by your laboratory should be of similar grade and granulation and the results should be recorded under sample (Spl) on the data sheet.

Although the samples are manufactured in powder form, caking frequently occurs. If this has happened, breakup all lumps before using, but do not reduce the particle size by grinding.

Sufficient amounts of each sample have been provided for practice runs so that preliminary estimates of the test times can be made.

Round Robin No. 24 **Data Sheet 1**

Determination of KI Heat Test Values for Inorganic Nitrates

Temp., °C	KI Test Time, Min.							
	Al		Co		Cu		Ni	
	Std	Spl	Std	Spl	Std	Spl	Std	Spl
65.5								
82.2								

Remarks:

Lab Code No. _____ Submitted By: _____

Round Robin No. 24 Data Sheet 2

Determination of MV Heat Test Values for Inorganic Nitrates

	MV Heat Test Values @ 120°C, Min.							
	Al		Co		Cu		Ni	
	Std	Spl	Std	Spl	Std	Spl	Std	Spl
Salmon Pink								
Red Fumes								

Lab Code No. _____ Submitted By: _____

Round Robin No. 24 **Data Sheet 3**

Determination of MV Heat Test Values for Inorganic Nitrates

MV Heat Test Values @ 134.5°C							
Al		Co		Cu		Ni	
Std	Spl	Std	Spl	Std	Spl	Std	Spl

Salmon Pink

Red Fumes

Lab Code No. _____ Submitted By: _____

Round Robin No. 24 **Data Sheet 4**

Specifications for Inorganic Nitrates
Supplied by Participating Laboratories

Compound	Specifications

Lab Code No. _____ Submitted By: _____

APPENDIX B

LABORATORY REMARKS SUBMITTED

LABORATORY REMARKS

Laboratory Code No. 1

The KI starch paper used in this test was manufactured at the U.S. Naval Powder Factory, Indian Head, Maryland, on 4 October 1955. Lot No. 191.

The 65.5°C KI test was conducted in an aluminum block heater, electrically heated, thermostatically controlled. The temperature was measured by a calibrated mercury thermometer.

The 82.2°C KI test was conducted in a steam heated 4/1 Ethyl Alcohol/Water bath; the temperature was measured by a calibrated mercury thermometer.

Lighting -- Transmitted incandescant lamp with a 150 watt daylight bulb, reflected off of a white curtain directly in back of the bath.

Viewing -- The bath was approximately at eye level and the observer's eyes were about one foot away directly in front of the bath.

Procedure -- Technical Report FRL-TR-25.

The methyl violet paper used in the 120° and 134.5°C methyl violet heat test was manufactured at theU.S. Naval Powder Factory, Indian Head, Maryland, on 22 June 1961. Lot No. 498.

The 120° and 134.5°C heat tests were conducted in an aluminum block heater, electrically heated, thermostatically controlled. The temperature was measured by a calibrated mercury thermometer.

Viewing -- The observer stands directly in front of the bath when lifting tubes to read end-points.

Lighting -- Aluminum block heater, enclosed in a hood opening to front only. Background is a white painted wall and a window. Transmitted fluorescent light from the room. No light directly on the aluminum block heater.

Procedure -- Technical Report FRL-TR-25.

Specifications for Inorganic Nitrates Supplied by Participating Laboratories

Compund	Granulation, % on No.			Specifications
Standard Samples	12	30	40	Fisher Catalog No.
$Al(NO_3)_3.9H_2O$	68	225	3	A-586
$Co(NO_3)_2.6H_2O$	34	65	1	C-378
$Cu(NO_3)_2.3H_2O$	33	54	7	C-467
$Ni(NO_3)_2.6H_2O$	6	78	11	N-62
Special Samples (Propellants Laboratory Stockroom)				
$Co(NO_3)_2.6H_2O$	Lot No. FD 51			Crystal Baker and Adamson Co.
$Al(NO_3)_2.9H_2O$	Lot No. 543057			Crystal Fischer Scientific Co.
$Ni(NO_3)_2.6H_2O$	Lot No. 54123			Crystal Fischer Scientific Co.
$Cu(NO_3)_2.3H_2O$	Lot No. 704802			Crystal Fischer Scientific Co.

Laboratory Code No. 2

Compound	J.T. Baker Lot No.	Granulation, % on No.		
		12	30	40
Al(NO3)3.9H2O	42949	95	5	--
Co(NO3)2.6H2O	112045	--	98	1
Cu(NO3)2.3H2O	1083	28	61	6

Laboratory Code No. 5

New bottle of Fisher C-467 $Cu(NO_3)_2.3H_2O$ hardened to a rock-like condition over night after being used on 23 March 1965. No additional tests were run.

Laboratory Code No. 6

Compound	Specifications
Aluminum Nitrate	Mallenckrodt Analyzed Reagent Grade approximately 5 years old
Nickel Nitrate	Baker, Analyzed Reagent approximately 2 years old
Copper Nitrate	Matheson, Coleman & Bell Reagent Grade, approximately 2 years old
Cobalt Nitrate	Fisher Scientific CP Grade, approximately 10 years old

Laboratory Code No. 7

Compound	Specifications	Fisher Catalog Number
Aluminum Nitrate, $Al(NO_3)_3.9H_2O$	Certified Grade	A-586
Cobaltous Nitrate, $Co(NO_3)_2.6H_2O$	Certified Grade	C-378
Copper(ic) Nitrate, $Cu(NO_3)_2.3H_2O$	Certified Grade	C-467
Nickel(ous) Nitrate, $Ni(NO_3)_2.6H_2O$	Certified Grade	N-62

Laboratory Code No. 11

Compound	Fisher Lot No.	Catalog No.	
$Al(NO_3)_3.9H_2O$	742725	A-586	3.0% retained on #12 sieve
$Co(NO_3)_2.6H_2O$	74536	C-378	21.8% retained on #12 sieve
$Cu(NO_3)_2.3H_2O$	733525	C-467	7.6% retained on #12 sieve
$Ni(NO_3)_2.3H_2O$	731828	N-62	44.2% retained on #12 sieve

Laboratory Code No. 12

KI Heat Test Values

Ni Std @ 65.5 did not melt.
Co Spl @ 65.5 did not melt.
NPP KI Starch Paper Lot 189

MV Heat Test Values

NPP normal violet paper Lot 10, 1964 - @ 120°C minute
NPP normal violet paper Lot 10, 1964 - @ 134.5°C minute
Chemical purchased from J. T. Baker Chemical Co.
$Cu(NO_3)_2.3H_2O$ contained foreign material.

Laboratory Code No. 13

It is fairly certain that the discoloration appeared before the entire sample attained the test temperature in many of these samples.

Compound	Fisher Catalog No.	Granulation, % on No.			
		#8	#12	#30	#40
$Al(NO_3)_3.9H_2O$	A-586	0.0	1.2	54.2	17.1
$Co(NO_3)_2.6H_2O$	C-378	15.0	17.4	46.7	11.2
$Cu(NO_3)_2.3H_2O$	C-467	9.8	21.2	63.0	2.8
$Ni(NO_3)_2.6H_2O$	N-62	29.8	24.1	44.7	1.4

Laboratory Code No. 14

Compound	Specification
Aluminum Nitrate	J. T. Baker #0528 Reagent Grade
Cobalt Nitrate	J. T. Baker #1680 Reagent Grade
Cupric Nitrate	J. T. Baker #1800 Reagent Grade
Nickelous Nitrate	J. T. Baker #2784 Reagent Grade

TABLE 1

KI HEAT TEST FOR $Al(NO_3)_3 \cdot 9H_2O$

Lab. No.	KI Test Time, Minutes															
	65.5°C								82.2°C							
	Standard				Sample				Standard				Sample			
	a	b	c	$\bar{x}$	a	b	c	$\bar{x}$	a	b	c	$\bar{x}$	a	b	c	$\bar{x}$
1	10	10	11	10	4	4	4.5	4	3	3	3	3	1.5	1.5	1.5	2
6	8	8	8	8	-	-	-	-	-	-	-	-	-	-	-	-
11	5	6	5	5	10	10	10	10	3	3	3.5	3	3.5	3.5	3.5	4
12	5	5	5	5	8	8	8	8	2	2	2	2	2	2	2	2
13	9	11	11	10	4	4	4	4	2	2	2	2	3	3	4	3
14	6	6	8	7	21	25	26	24	3	3	3	3	2	2	2	2

APPENDIX C

TABLES

TABLE 2

KI HEAT TEST FOR $Co(NO_3)_2 \cdot 6H_2O$

Lab No.	KI Test Time, Minutes															
	65.5°C								82.2°C							
	Standard				Sample				Standard				Sample			
	a	b	c	$\bar{x}$	a	b	c	$\bar{x}$	a	b	c	$\bar{x}$	a	b	c	$\bar{x}$
1	60+	60+	60+	60+	60+	60+	60+	60+	54	54	54	54	60+	60+	60+	60+
6	514	514	514	514	--	--	--	--	--	--	--	--	--	--	--	--
11	60+	60+	60+	60+	60+	60+	60+	60+	60	60	60	60	20	20	20	20
12	60+	60+	60+	60+	60+	60+	60+	60+	60+	60+	60+	60+	60+	60+	60+	60+
13	38	49	52	46	12	14	16	14	10	10	12	11	6	6	7	6
14	60+	--	--	60+	60+	60+	60+	60+	13	13	15	14	57	58	59	58

TABLE 3

KI HEAT TEST FOR $Cu(NO_3)_2 \cdot 3H_2O$

Lab No.	KI Test Time, Minutes															
	65.5°C								82.2°C							
	Standard				Sample				Standard				Sample			
	a	b	c	$\bar{x}$	a	b	c	$\bar{x}$	a	b	c	$\bar{x}$	a	b	c	$\bar{x}$
1	9	10	10	10	2	2	2	2	4	4	4	4	1.5	1.5	1.5	2
6	7	7	7	7	-	-	-	-	-	-	-	-	-	-	-	-
11	6	6	5	6	1	1	2	2	4	4	4	4	2	2	2	2
12	5	5	5	5	6	6	6	6	2	2	2	2	2	2	2	2
13	5	6	6	6	2	2	2	2	1	1	1	1	1	1	1	1
14	7	9	9	8	8	10	11	10	5	5	5	5	5	6	6	6

TABLE 4

KI HEAT TEST FOR $Ni(NO_3)_2 \cdot 6H_2O$

Lab No.	KI Test Time, Minutes															
	65.5°C								82.2°C							
	Standard				Sample				Standard				Sample			
	a	b	c	$\bar{x}$	a	b	c	$\bar{x}$	a	b	c	$\bar{x}$	a	b	c	$\bar{x}$
1	7	7	7	7	6	6	6	6	5	6.5	8.5	7	4	4	4	4
6	124	124	124	124	-	-	-	-	--	--	--	--	-	-	-	-
11	60+	60+	60+	60+	1.5	1	1	1	17	17	17	17	0.5	0.5	0.5	1
12	60+	60+	60+	60+	32	32	32	32	12	12	12	12	13	13	13	13
13	27	33	33	34	1	1	1	1	6	6	7	6	1	1	1	1
14	44	--	--	44	60+	60+	60+	60+	11	11	11	11	4	5	5	5

TABLE 5

MV HEAT TEST FOR $Al(NO_3)_3 \cdot 9H_2O$

Lab No.	Salmon Pink Test Time, Minutes															
	120°C								134.5°C							
	Standard				Sample				Standard				Sample			
	a	b	c	$\bar{x}$	a	b		$\bar{x}$	a	b	c	$\bar{x}$	a	b	c	$\bar{x}$
1	20	20	20	20	15	15	15	15	20	20	20	20	15	15	15	15
2	25	25	25	25	30	30	30	30	25	25	25	25	25	25	25	25
5	16	16	16	20	15	15	15	15	--	--	--	--	--	--	--	--
6	19	19	19	20	--	--	--	--	12	12	12	15	--	--	--	--
7	15	15	15	15	15	15	15	15	5	5	5	5	5	5	5	5
12	17	17	17	20	16	20	20	20	12	12	12	15	10	10	10	10
13	15	15	20	20	15	15	20	20	15	15	15	15	10	10	10	10
14	10	15	10	15	10	10	10	10	10	15	10	15	10	10	10	10

TABLE 6

MV HEAT TEST FOR $Co(NO_3)_2 \cdot 6H_2O$

Lab No.	Salmon Pink Test Time, Minutes															
	120°C								134.5°C							
	Standard				Sample				Standard				Sample			
	a	b	c	$\bar{x}$	a	b	c	$\bar{x}$	a	b	c	$\bar{x}$	a	b	c	$\bar{x}$
1	230	260	300+		220	265	300+		85	90	100		85	90	100	
2	300+	300+	300+		200	190	195		40	45	45		40	40	45	
5	300+	300+	300+		---	---	---		--	--	--		--	--	--	
6	249	259	300+		---	---	---		125	135	138		--	--	--	
7	180+	180+	180+		135	135	135		50	50	50		60	60	60	
12	168	203	203		300+	300+	300+		94	121	164		111	122	125	
13	300+	300+	300+		210	200	220		110	115	125		140	155	170	
14	260	300	270		25	260	270		80	130	65		105	100	85	

TABLE 7

MV HEAT TEST FOR $Cu(NO_3)_2 \cdot 3H_2O$

Lab No.	Salmon Pink Test Time, Minutes															
	120°C								134.5°C							
	Standard				Sample				Standard				Sample			
	a	b	c	$\bar{x}$	a	b	c	$\bar{x}$	a	b	c	$\bar{x}$	a	b	c	$\bar{x}$
1	20	20	20	20	15	15	15	15	15	15	15	15	10	10	10	10
2	30	30	30	30	30	30	30	30	15	15	20	20	15	15	15	15
5	14	14	14	15	17	21	21	20	--	--	--	--	--	--	--	--
6	17	17	18	20	--	--	--	--	13	13	14	15	--	--	--	--
7	15	15	15	15	15	15	15	15	5	5	5	5	5	5	5	5
12	19	19	20	20	22	22	22	25	11	11	11	15	9	9	11	10
13	15	15	15	15	15	15	15	15	15	15	15	15	10	10	10	10
14	15	20	15	20	15	15	15	15	10	15	10	15	15	15	15	15

TABLE 8

MV HEAT TEST FOR $Ni(NO_3)_2 \cdot 6H_2O$

Lab No.	Salmon Pink Test Time, Minutes															
	120°C								134.5°C							
	Standard				Sample				Standard				Sample			
	a	b	c	$\bar{x}$	a	b	c	$\bar{x}$	a	b	c	$\bar{x}$	a	b	c	$\bar{x}$
1	35	70	100		120	120	130		35	45	45		50	50	60	
2	165	165	170		---	---	---		45	45	45		--	--	--	
5	100	105	125		---	---	---		--	--	--		--	--	--	
6	99	109	117		---	---	---		44	44	46		--	--	--	
7	90	90	90		55	55	55		30	30	30		25	25	25	
12	100+	100+	100+		100+	100+	100+		46	60	60		46	63	86	
13	135	135	140		45	45	45		50	50	50		30	45	55	
14	120	100	100		200	175	180		50	55	50		100	95	100	

TABLE 9

MV HEAT TEST FOR Al $(NO_3)_3 \cdot 9H_2O$

Lab No.	Red Fumes Test Time, Minutes															
	120°C								134.5°C							
	Standard				Sample				Standard				Sample			
	a	b	c	$\bar{x}$	a	b	c	$\bar{x}$	a	b	c	$\bar{x}$	a	b	c	$\bar{x}$
1	20	20	20	20	15	15	15	15	15	15	15	15	10	10	10	15
2	25	25	25	25	30	30	30	30	15	15	15	15	15	15	15	15
5	16	16	16	16	15	15	15	15	--	--	--	--	--	--	--	--
6	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
7	20	20	20	20	20	20	20	20	10	10	10	10	10	10	10	10
12	20	20	20	20	20	20	20	20	14	14	14	14	11	11	12	12
13	20	20	20	20	20	20	20	20	15	15	15	15	10	10	10	10
14	20	20	20	20	20	20	20	20	10	15	10	15	10	10	10	19

TABLE 10

MV HEAT TEST FOR $Co(NO_3)_2.6H_2O$

Lab No.	Red Fumes Test, Time, Minutes															
	120°C								134.5°C							
	Standard				Sample				Standard				Sample			
	a	b	c	$\bar{x}$	a	b	c	$\bar{x}$	a	b	c	$\bar{x}$	a	b	c	$\bar{x}$
1	300+	300+	300+		245	285	300+		300+	300+	300+		175	180	180	
2	300+	300+	300+		300+	300+	300+		200	205	210		160	160	160	
5	300+	300+	300+		---	---	---		---	---	---		---	---	---	
6	---	---	---		---	---	---		---	---	---		---	---	---	
7	300+	300+	300+		300+	300+	300+		180+	180+	180+		180+	180+	180+	
12	400+	400+	400+		300+	300+	300+		300+	300+	300+		118	132	132	
13	300+	300+	300+		300+	300+	300+		300+	300+	300+		300+	300+	300+	
14	300+	300+	300+		300+	300+	300+		260	280	300+		280	180	300+	

TABLE 11

MV HEAT TEST FOR $Cu(NO_3)_2 \cdot 3H_2O$

Lab No.	Red Fumes Test Time, Minutes															
	120°C								134.5°C							
	Standard				Sample				Standard				Sample			
	a	b	c	$\bar{x}$	a	b	c	$\bar{x}$	a	b	c	$\bar{x}$	a	b	c	$\bar{x}$
1	25	25	25	25	15	15	15	15	15	15	15	15	15	15	15	15
2	30	30	30	30	30	30	30	30	15	15	15	15	15	15	15	15
5	16	16	16	16	--	--	--	--	--	--	--	--	--	--	--	--
6	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
7	20	20	20	20	20	20	20	20	10	10	10	10	10	10	10	10
12	19	19	19	19	22	22	22	22	16	17	17	17	16	16	16	16
13	20	20	20	20	20	20	20	20	20	20	20	20	15	15	15	15
14	20	20	20	20	20	15	20	20	15	15	15	15	15	15	15	15

TABLE 12

MV HEAT TEST FOR $Ni(NO_3)_2 \cdot 6H_2O$

Lab No.	Red Fumes Test Time, Minutes															
	120°C								134.5°C							
	Standard				Sample				Standard				Sample			
	a	b	c	$\bar{x}$	a	b	c	$\bar{x}$	a	b	c	$\bar{x}$	a	b	c	$\bar{x}$
1	45	85	120	---	120	120	130	---	100	105	105	---	50	50	60	---
2	300+	300+	300+	---	---	---	---	---	240	230	240	---	---	---	---	---
5	280	280	280	---	---	---	---	---	---	---	---	---	---	---	---	---
6	---	---	---	---	---	---	---	---	240	300	---	---	---	---	---	---
7	180+	180+	180+	---	180+	180+	180+	---	180+	180+	180+	---	180+	180+	180+	---
12	300+	300+	300+	---	300+	300+	300+	---	50	104	104	---	104	104	104	---
13	300+	300+	300+	---	300+	300+	300+	---	300+	300+	300+	---	300+	300+	300+	---
14	300+	300+	300+	---		300+	300+	---	260	250	26	---	270	180	280	---

TABLE 13

SUMMARY OF KI AND MV TEST RESULTS FOR Al AND Cu

Lab No.	KI Test Time, Minutes								MV (Salmon Pink) Test Ti , Minutes								MV(Red Fumes) Test Time, Minutes							
	65°C				82.2°C				120°C				134.5°C				120°C				135.5° C			
	Al		Cu		Al		Cu		Al		Cu		Al		Cu		Al		Cu		Al		Cu	
	Std	Spl	Std	Spl	Std	Spl	Std	Spl	Std	Spl	Std	Spl	Std	Spl	Std	Spl	Std	Spl	Std	Spl	Std	Spl	Std	Spl
1	10	4	10	2	3	2	4	2	20	15	20	15	20	15	15	10	20	15	25	15	15	15	15	15
2	--	--	--	--	-	-	-	-	25	30	30	30	25	25	20	15	25	30	30	30	15	15	15	15
5	--	--	--	--	-	-	-	-	20	15	20	15	--	--	--	--	16	15	16	--	--	--	--	--
6	8	--	7	--	-	-	-	-	24	--	20	--	15	--	15	--	--	--	--	--	--	--	--	--
7	--	--	--	--	-	-	-	-	15	15	15	15	5	5	5	5	20	20	20	20	10	10	10	10
11	5	10	6	2	3	4	4	2	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
12	5	8	5	6	2	2	2	2	20	20	20	25	15	10	15	10	20	20	19	22	14	12	17	16
13	10	4	6	2	2	3	1	1	20	20	15	15	15	10	15	10	20	20	20	20	15	10	20	15
14	7	24	8	10	3	2	5	6	15	10	20	15	15	10	15	15	20	20	20	20	15	10	15	15
$\bar{X}$	8	10	7	4	3	3	3	3	19	18	20	19	16	13	15	11	--	--	--	--	--	--	--	--
s	2.3	8.2	1.8	3.6	0.7	1.0	1.7	2	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--

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The data from this round robin indicates that aluminum nitrate and copper nitrate are suitable for use as heat test standards. These inorganic compounds are preferable to the organic nitrates currently being used because of greater stability, safety, purity and availability. These findings were reported at the 22nd meeting of the Interagency Chemical Rocket Propulsion Group (ICRPG) on Analytical Chemistry at the NASA-Lewis Research Center in Cleveland on 3-5 November 1965.

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